Trap Sample Splitting (wet):

Use of Sediment Traps for the Measurement of Particle and Associated Contaminant Fluxes

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Flux is equal to the mass collected divided by the length of collection and the trap cross section. In order to calculate fluxes from the trapped material a reliable measurement of the total weight is required. In previous studies we had always split sediment trap samples after they were freeze dried and weighed. Pat VanHoof, who will be analyzing these samples for PCBs and other trace organic contaminants, wants to extract all of her samples while they are still wet. In splitting the sample while wet, it is necessary to be able to estimate the total weight of the sample from some fraction of that material.

Thus it was necessary to buy or develop a wet sample splitting procedure. A wet splitter for trap samples, designed at Woods Hole, is commercially available for $6-7000 and it splits samples into four or eight subsamples. This was both too expensive and fractionated the samples too much; we would need to recombine to get our two fractions requiring considerable container cleaning, etc. as excess overhead.

After further literature and catalog searches we purchased an all stainless steel dry sediment sample micro-splitter (Model SP-241x; Gilson Co. Inc., PO Box 677, Worthington, OH, 43085-0677). This device has a reservoir of approximately 80 mL into which the sample is poured. A bottom vent is then opened and the sample pours into 30 evenly spaced (1 mm) slots. The even numbered slots empty into a stainless steel tray on the left and the odd numbered slots empty on the right. We then tested this device for our wet sample splitting requirements and came up with satisfactory results, described below.

Sample Matrices: We examined four samples. The objective was to determine the precision of splitting and the ratio of the two samples. The four samples were:

1. Distilled water (DDW)

2. Distilled water (55 mL) + chloroform (6 mL); our standard trap poison solution

3. Ground Lake Michigan sediment in # 2

4. A sediment trap sample from Lake Michigan near LMMB station 6; 5m above bottom from a 100m deep station.

Five replicates of each matrix were made. The samples were poured into the splitter and the left and right trays weighed for matrices 1 and 2. For matrices 3 and 4, the left and right trays were emptied into preweighed beakers which were dried at 90°C then weighed. The data are presented in Table 1.

Table 1. Sample Splitting Data

	Total Dry Wt (g)	Wt (left) (g)	Wt (Right) (g)	Fract left	Fract Rt
DDW		33.4473	31.4184	0.516	0.484
DDW		32.5575	30.962	0.513	0.487
DDW		32.9653	30.9628	0.516	0.484
DDW		32.2945	29.296	0.524	0.476
DDW		31.7108	29.3542	0.519	0.481
DDW(55):CHCl3(6)		31.6683	33.0099	0.490	0.510
DDW(55):CHCl3(6)		30.2318	31.3103	0.491	0.509
DDW(55):CHCl3(6)		31.2056	31.5524	0.497	0.503
DDW(55):CHCl3(6)		30.8368	31.6704	0.493	0.507
DDW(55):CHCl3(6)		31.0031	33.3368	0.482	0.518
Grnd Sed in DDW(55):CHCl3(6); DRY	0.5639	0.2779	0.286	0.493	0.507
Grnd Sed in DDW(55):CHCl3(6); DRY	1.387	0.6952	0.6918	0.501	0.499
Grnd Sed in DDW(55):CHCl3(6); DRY	2.9349	1.5035	1.4314	0.512	0.488
Grnd Sed in DDW(55):CHCl3(6); DRY	3.9479	1.9049	2.043	0.483	0.517
Grnd Sed in DDW(55):CHCl3(6); DRY	5.1343	2.5843	2.55	0.503	0.497
Trap from 5m AB @ 100 m sta.; DRY	0.4434	0.2224	0.221	0.502	0.498
Trap from 5m AB @ 100 m sta.; DRY	0.7476	0.367	0.3806	0.491	0.509
Trap from 5m AB @ 100 m sta.; DRY	1.2745	0.6423	0.6322	0.504	0.496
Trap from 5m AB @ 100 m sta.; DRY	1.3124	0.648	0.6644	0.494	0.506
Trap from 5m AB @ 100 m sta.; DRY	2.2998	1.1689	1.1309	0.508	0.492

Excellent replication was obtained in the tests (Table 2). Matrices 3 and 4, with sediment or trap materials, were split into two equal portions without bias. In other studies we have determined that replicate traps placed side by side have a coefficient of variation (100*sd/mean) of a little less than 10%. The splitting errors appear substantially smaller and will not degrade our interpretation of the data.

Table 2. Accuracy and precision of sample splitting (n=5; all mixtures)

Mixture	Left Side Fraction	Right Side Fraction	P (paired t)
DDW	0.518 ± 0.004	0.483 ± 0.004	
DDW + CHCl3	0.491 ± 0.005	0.509 ± 0.005	
Ground sediment	0.501 ± 0.001	0.499 ± 0.001	0.92
Ground sediment Org C	6.68 ± 0.01	6.62 ± 0.02	0.56
Trap	0.500 ± 0.006	0.500 ± 0.006	0.93

Our standard splitting procedure will be:

1. Allow the 60 mL trap bottles to settle for approximately 24 hours in refrigeration.
2. Pour off approximately 25 mL of the overlying water into a pre-cleaned beaker.
3. Pour the remaining trap sample through a 700 µm screen into the splitter reservoir.
4. Split by opening the bottom valve.
5. Rinse with the water from #2.
6. Further rinse (if needed) with pre-extracted DDW.
7. Pour left tray back into trap sample bottle for freeze drying.
8. Pour right side into pre-cleaned glass jar for PCB, etc.
9. Transfer >700 µm materials to precleaned, preweighed scintillation vial.
10. Rinse screen and splitter under faucet, then with pre-extracted DDW.